GRScientific

Aquamax KF Volumetric

USER MANUAL

GUARANTEE

The **Aquamax KF Volumetric** titrators are guaranteed against any manufacturing defect or component failure.
GRScientific will repair or replace, free of charge, any defective component or parts of the instruments under guarantee.
This guarantee does not cover damage caused by accident, improper use or internal handling by unauthorised persons.

Syringes and valves will be replaced under guarantee once our after sales service confirms they have a factory defect.
This guarantee does not cover defects caused by:
- incorrect use,
- damage caused by accidents,
- premature wear caused by certain working conditions.

Electrode is also guaranteed against manufacturing defects.
GRScientific will replace any sensor deemed to have factory defects free of charge, once confirmed by our After Sales Service.
This guarantee does not cover defects caused by:
- incorrect use,
- normal sensor wear,
- logical premature wear caused by certain samples,
- damage caused by accidents.

Validity

Instrument: 5 years.
Sensors: 6 months

In the event of failure

Contact your local GRScientific dealer.

The continuous improvement of our instruments can bring about differences between the information written in this manual and the purchased instrument.
The data, figures and descriptions contained in this manual cannot be legally asserted.
GRScientific reserves the right to make changes and corrections without prior notice.

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DECLARATION OF CONFORMITY CE

Declaration of conformity

We — **GRScientific**
P.O. BOX 242, Ampthill, Bedfordshire
MK45 5AQ. UK
declare under our sole responsibility that the product,

Description — **Aquamax KF Volumetric**

to which this declaration relates is in conformity with the following normative documents:

Low-voltage directive — **2006/95/CE**

Standard	UNE-EN 61010-1	07-2002
	UNE-EN 61010-1	11-2003

EMC Directive — **2004/108/CE**

Standard	UNE-EN 61326-1	10-2006

Place and date — **Ampthill, 06.07.2009**

GRScientific

Edition: July 2009

SAFETY MEASURES

Warning

Please, read carefully this information before installing and using the instrument.

These safety measures do not purport to address all safety problems associated with the use of this instrument.
It is the responsibility of whoever uses this instrument to consult and establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1. Never work in an environment subject to explosion hazards. The housing of the instrument is not gas tight.
2. Using chemicals, comply with the general instructions for hazard prevention and safety rules, e.g. wear protective clothing, eye protection and gloves.
3. Read carefully the manual of the instrument.
4. In case of liquids splashes, wipe off immediately. The instrument is not waterproof.
5. Exclude the following environmental influences:
- vibrations
- direct sunlight
- atmospheric humidity higher than 80 %
- corrosive gases present
- temperature below 15 ºC and above 40 ºC
- powerful electric and magnetic fields.
6. Only use original accessories and spare parts.
7. Have the instrument serviced only by GRScientific Service.

PRESENTATION

The Aquamax KF Volumetric is for volumetric determination of water, using Karl Fischer method. It operates with any type of KF reagents (preferable without pyridine) and are suitable for determination from few ppm up to 100% of H_2O.

The messages on the titrator display guide the user making the instrument suitable for routine analysis.

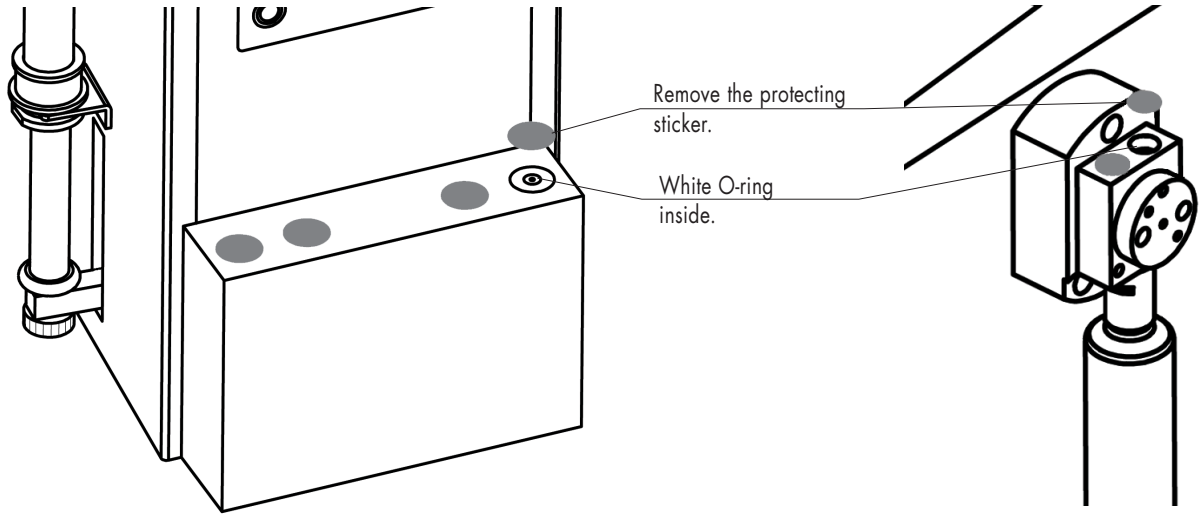
Supplied accessories

Aquamax KF Volumetric

1 x Plug in power supply 90-264 VAC, 47-63 Hz, 24 Volt DC, code 91 87 36.
2 x TLL SL syringe, 5 ml, code 91 92 28.
2 x Burette intlet tube with bottle cap, DIN 45 screw, code 91 87 08.
2 x Burette outlet tube with antidiffuser device, code 91 87 01.
5 x drier cartridges with molecular sieve, code 91 92 00.
1 x Inlet pump tube (for solvent dispensing) with bottle cap DIN 45 screw, code 91 87 05.
1 x Outlet tube with conical adapter for solvent dispensing pump, code 91 87 10.
1 x Inlet tube with conical adapter for emptying vessel pump, code 91 87 10.
1 x Outlet pump tube (for vessel emptying) with bottle cap DIN 45 screw, code 91 87 29.
1 x Support for electrode and tubes with magnetic stirrer and vessel for KF titration, code 91 91 60.
1 x Double platinum electrode, code 91 52 64.
1 x Electrode cable with BNC connector, code 91 90 55.
1 x Pack of white O-rings (10 units) for valves and pumps screws, code 91 87 34.

Optional accessories

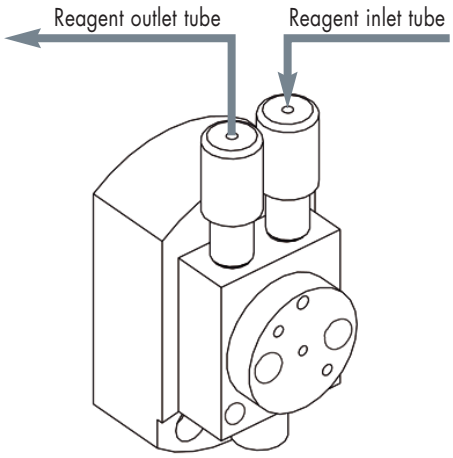
Thermal printer, code 91 82 01.
Dot-matrix printer, code 91 82 00
Standard PC keyboard, code 91 90 13.
Communication software TiCom, code 91 86 82.



1. Remove the protecting stickers from the inlet and outlet of the pumps and the valves.
2. Check that there is a small white O-ring in every screw. It must be in horizontal position. This O-ring ensures the union tightness.

⚠ **Attention**: If the O-ring is not inside, check that it is not stuck on the protecting sticker.

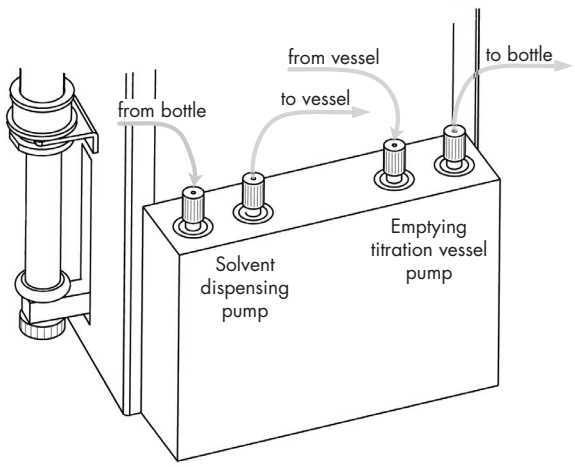
... of the burette tubes



1. Screw the reagent outlet tube on the position placed on the left side of the valve. Place the tube on the sensor support.
2. Screw the reagent inlet tube on the position placed on the right side of the valve. Introduce the tube into the reagent bottle and screw the cap.

Important: In order to avoid possible reagent leakage, it is strongly advisable to periodically check and re-tighten the screw joints, especially during the first days of use of the Aquamax KF Volumetric.

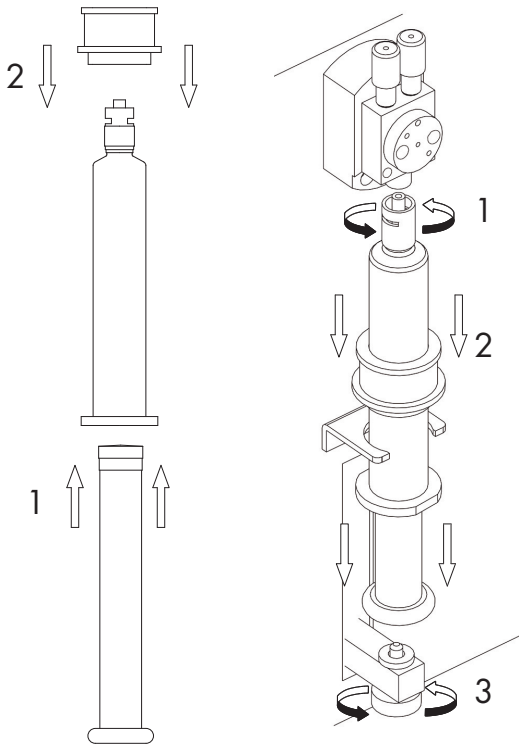
... of the pumps tubes

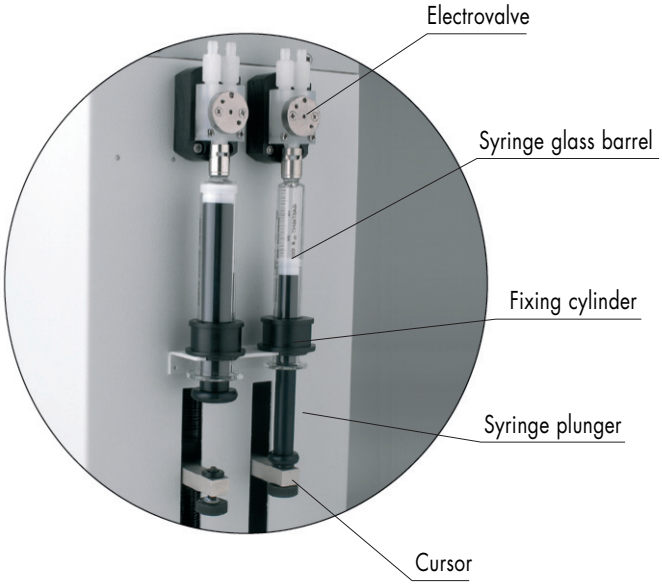


Important: In order to avoid possible reagent leakage, it is strongly advisable to check and re-tighten periodically the screw joints, especially during the first days of use of the Aquamax KF Volumetric.

INSTALLATION

... of the syringe



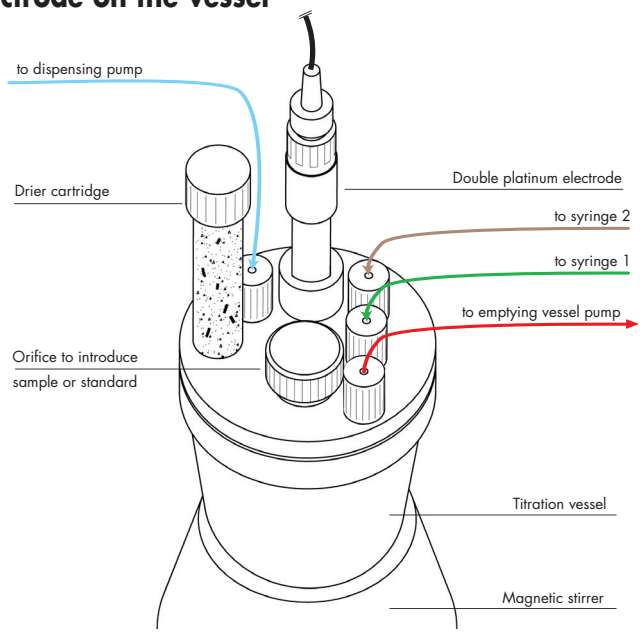


In the Aquamax KF Volumetric, the two syringes are activated via a single mechanism.
The plunger of the syringes must be detached to the cursor. The cursor in movement drags the plunger of the attached syringes.
Only one reagent is dispensed, the corresponding to the activated valve. The other syringe will return the reagent to the original bottle.

To install the syringe follow the described procedure:
A. Introduce 1/3 part of the plunger in the glass barrel. Put the fixing cylinder as shown.
B. Switch on the instrument. The cursors automatically go to down position.
C. Screw the syringe on the valve (1). Place the fixing cylinder on the metal clamp (2). Pull the plunger if required and screw it on the syringe plunger (3).

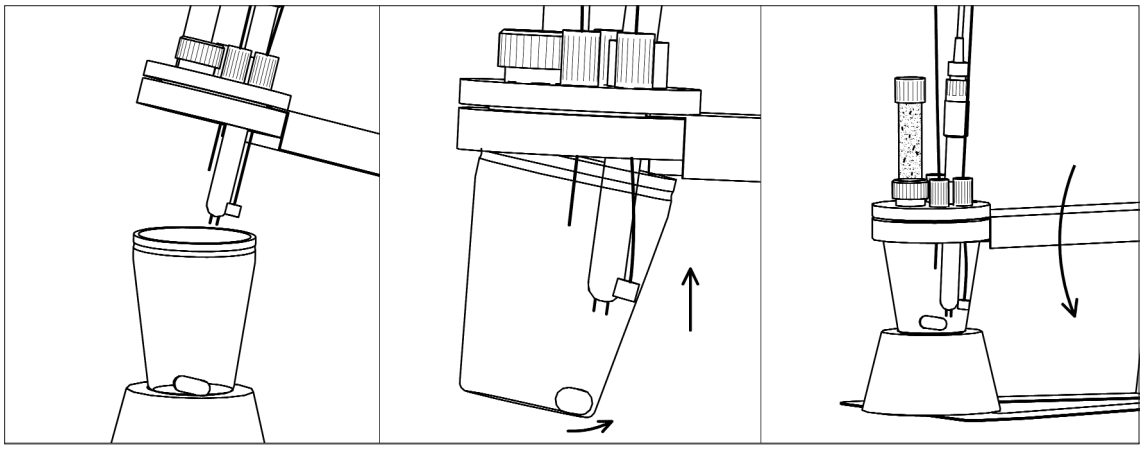
Important: In order to avoid possible reagent leakage, it is strongly advisable to periodically check and re-tighten the syringe in the valve, especially during the first days of use of the Aquamax KF Volumetric.

... of the tubes and the electrode on the vessel



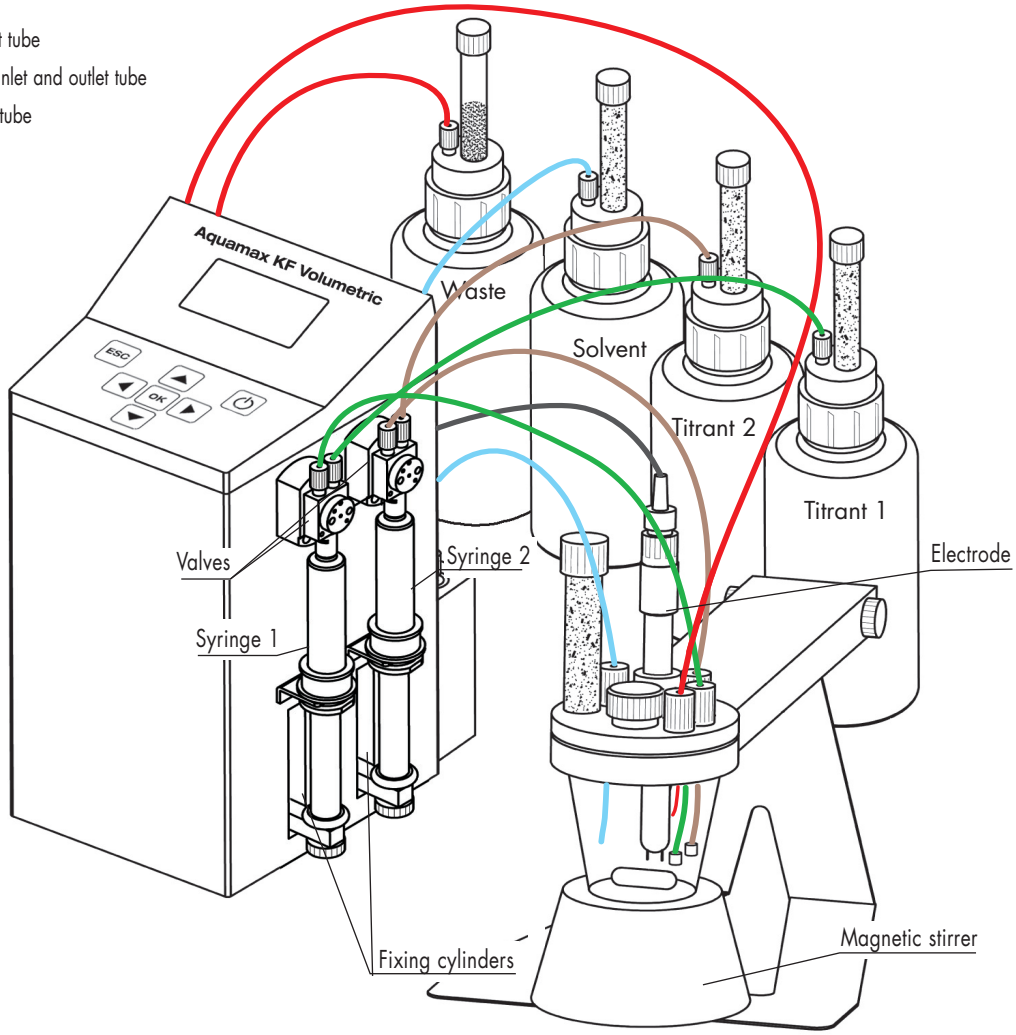
INSTALLATION

... of the titration vessel



GENERAL VIEW

- Electrode cable
- Titrant reagent 1, inlet and outlet tube
- Titrant reagent 2 (or Standard), inlet and outlet tube
- Solvent reagent, inlet and outlet tube
- Waste tubes



The syringe on the left corresponds to the Reagent 1 (R1), meanwhile the syringe on the right corresponds to the Reagent 2 (R2).
Connect the plug-in power supply, electrode cable and magnetic stirrer to the rear panel.

Keypad

On/Off.

ESC Move backwards in the menus.
Interrupt a Manual actuation.
Interrupt a titration.
When starting a calibration, it displays the last calibration data.

OK Move forwards in the menus confirming the option marked in gray (in bold in this manual).
In the data-logger menu it displays the first register.

▲ Selection between the different options displayed.
Selection of numeric values.
In the Data-logger it displays the former register.
In Manual actuation it empties the syringe through the outlet tube.

▼ Selection between the different options displayed.
Selection of numeric values.
In the Data-logger it displays the following register.
In Manual actuation it loads the syringe through the inlet tube.

▶ Move forwards a digit when entering a numeric value.
Show the next display in those menus that have more than one.

◀ Move backwards a digit when entering a numeric value.
Show the previous display in those menus that have more than one.
In Manual actuation it empties the syringe through the inlet tube.

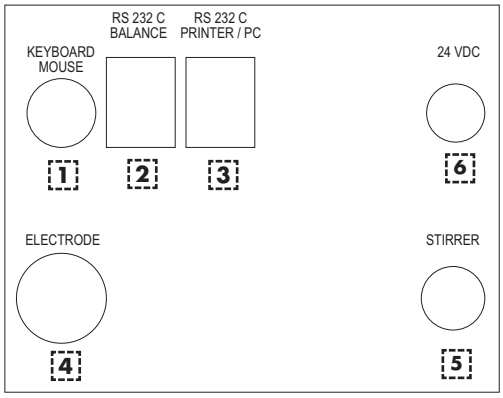
External keyboard: Standard PC keyboard

Its main use is the text introduction: header, user name, reagent name, and alphanumeric sample identification. It allows the introduction of numeric data too.
If the automatic sample identification is used, it is not necessary the connection of a PC keyboard to the Aquamax KF Volumetric.

⚠ Precaution

The external keyboard always must be connected before switching on the instrument.

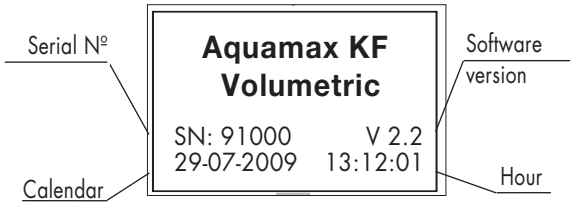
Rear panel



Connections for:
1, External keyboards. 2, Balance. 3, Printer or PC.
4, Electrode. 5, Magnetic stirrer. 6, Plug in power supply.

Stand-by screen

If the instrument is not used for more than 5 minutes it will automatically display the "Stand-by" screen.
Press any key to go back to the previous screen.



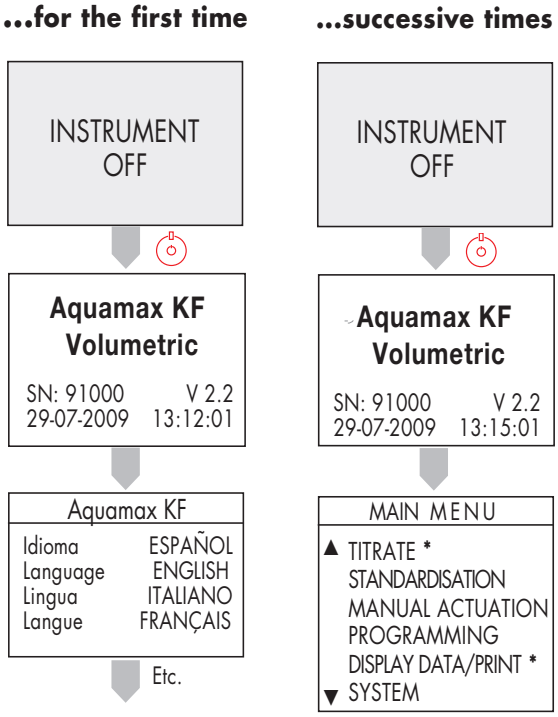
START UP

When switching on the instrument for the first time must be selected:
Language.
2nd syringe function. Select the function between:
Standard dispensing
Titrate
Data output. Selecting "Printer" or "Computer", the Aquamax KF Volumetric will ask for:
- Report header . Two lines of 20 characters for company name introduction, etc. The use of a PC keyboard is necessary.
- Sample ID. "Manual", sample identification introduction per each sample; "Automatic", consecutive sample identification assigned by the titrator.
The instrument presents the Main Menu.

From the option SYSTEM in the main menu, the user can select: weight entrance, date and hour, user´s name and reagent´s name (see page 18).

⚠ **Precaution**
Before titration, load the syringes with the corresponding reagents, see page 12.

Switch on...



* If the 2nd syringe is configured for titration, in the main menu will appear "TITRATE R1, TITRATE R2"

** The option PRINT appears only if a printer is connected.

FACTORY CONFIGURATION

2nd syringe is configured for standard dispensing:

The instrument has 4 titration programs and 1 standardization program per syringe nº1. The factory setting is the following:

Reagent R1:
Program P1: KF Standard: Activated.
Titration type: Classic.
Drift control: automatic, drift limit: 30 µl/min.
Units: %.
Program 2: KF < 1% H_2O. Deactivated.
Program 3: KF > 10% H_2O. Deactivated.
Program 4: KF Standard. Deactivated.
Standardization program: Classic.
Water standard: Water standard in volume.

2nd syringe is configured for titration:

The instrument has 4 titration programs and 1 standardization program per every syringe. The factory setting is the following:

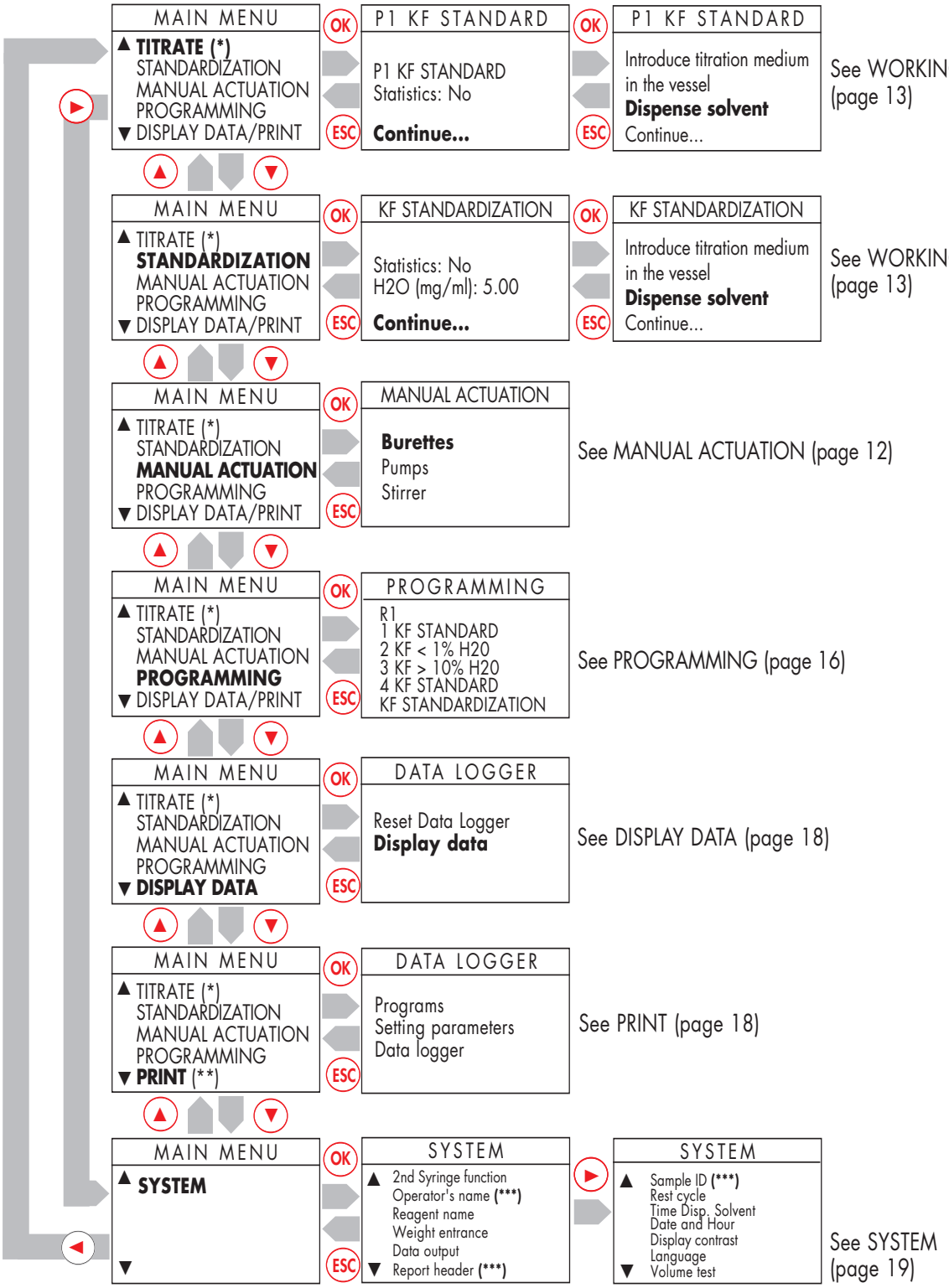
Reagent R1:
Program P1: KF Standard: Activated.
Titration type: Classic.
Drift control: automatic, drift limit: 30 µl/min.
Units: %.
Program 2: KF < 1% H_2O. Deactivated.
Program 3: KF > 10% H_2O. Deactivated.
Program 4: KF Standard. Deactivated.
Standardization program: Classic.
Water standard: Water standard in volume.

Reagent R2:
The same configuration as the Reagent R1.

Activated Program: An activated program can be selected to perform titration, to be modified or visualized.

Deactivated Program: A deactivated program does not appear as an option to perform titration. It is recommended to deactivate the programs which usually will not be used.
When activating / deactivating a program the programmed parameters do not modify.

To modify the programms or activate a new program, see PROGRAMMING, page 16.



* If the 2nd syringe is configured for titration, the main menu will show TITRATE R1 and TITRATE R2.

** The option PRINT will only appear if a printer is connected.

*** These options will only appear if Data Output is activated.

MANUAL ACTUATION

Syringe

The reagent inlet and outlet tubes must be correctly installed to avoid accidental spillage of reagent outside the titration vessel or the reagent bottle. Before any titration the liquid circuit must be primed with the titrant reagent.

Select the option Manual Actuation, Burette and press the keys corresponding to Fill (the syringe fills with reagent from the bottle) and Empty (empty the syringe content into the titration vessel). It is recommended to repeat this process several times until the liquid circuit is completely primed.

In the Aquamax KF Volumetric the two syringes can be primed simultaneously. For this purpuse select both reagents. When only one syringe is selected, the other fills and returns the reagent in the original bottle.

Important: When the syringe piston reaches the position up or by pressing the key ESC, the syringe movement is interrupted. When one of the syringes is not selected, the reagent is aspirated and returned to the bottle.

Return reagent:

This options allows to return the syringe content into the bottle. It is recommended at the end of the working session, before cleaning or changing the reagent.

Pumps

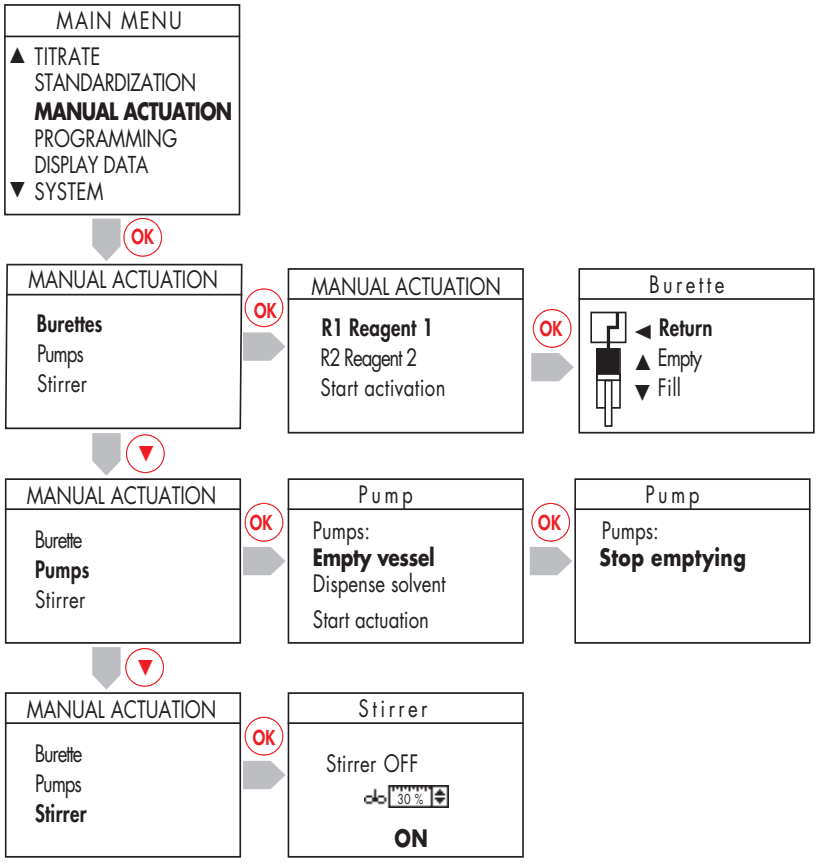
The reagent inlet and outlet tubes must be correctly installed to avoid accidental spillage of reagent outside the titration vessel or the reagent bottle.

The pump 1 is configured for solvent dispensing. Hence the inlet tube (IN) must be installed in the reagent bottle and the outlet tube (OUT) must be installed on the titration vessel lid, see page 6.

The pump 2 is configured for vessel emptying. Hence the inlet tube (IN) must be installed on the titration vessel and the outlet tube (OUT) must be installed in the waste bottle, see page 6.

Stirrer

This option allows to activate the stirrer. Once activated the stirring speed can be modified.



WORKING PROCEDURE

The most common working procedure consists of reagent standardization followed by sample analysis.

Factor determination of titrant reagent (standardization)

A titration allowing to determine the titre of the titrant reagent, called Factor, expressed in mg of H2O/ml.
When the two syringes are configured for titration, select the reagent to be standardised, Reagent 1 or Reagent 2.

1. Data entrance

Statistics. To obtain statistical calculations, the number of analysis for the Factor determination must be introduced. As results the main value, the standard deviation and the relative standard deviation are presented.
Water content in the used standard. Introduce it according to the certificate of analysis of the standard.
Identification of the user. This option appears only when the data output is activated.

2. Introduction of the titration medium

In the Aquamax KF Volumetric it is enough to press the key OK when the message "Dispense Solvent" appears and automatically the required solvent volume is dispensed into the titration vessel. See page 19.
It should cover the two platimun wires of the electrode and the tips of the two burettes. See page 15.

3. Pre-titration and vessel conditioning

The Aquamax KF Volumetric neutralises the water present in the solvent and automatically activates the drift control. On the display appears the message *WAIT* until the vessel is conditioned. Hence the message "Continue" appears.

4. Water Standard Introduction

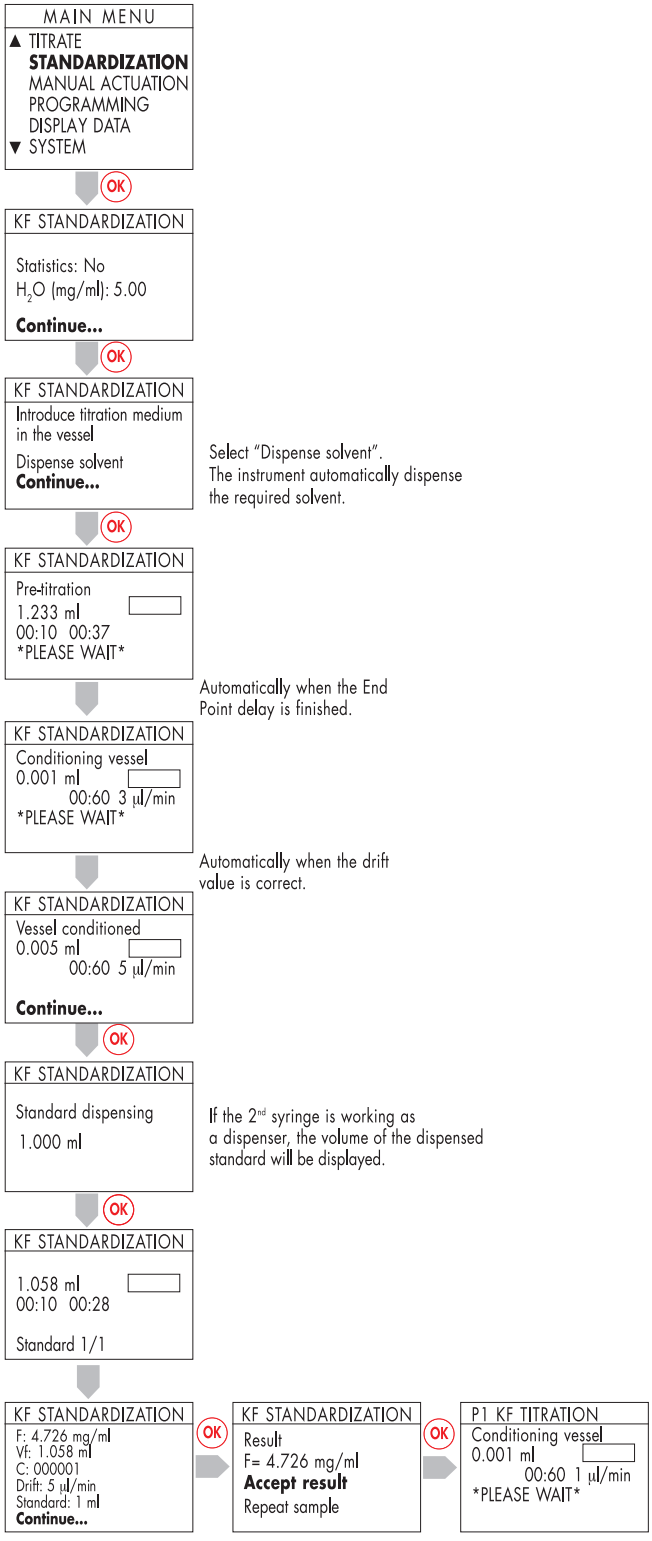
Introduce the standard into the titration vessel and start the standardization.
When the 2nd syringe is configured as dispensing syringe and a standard in volume is used, it is introduced automatically.

5. Result and Drift Control

Once the analysis has finished the Aquamax KF Volumetric activates automatically the drift control. The result is shown on display or sent to the PC or printer.
If there are statistical calculations the Aquamax KF Volumetric allows repeating the results considered as incorrect.

6. Factor storage

When the result is accepted, the factor is stored automatically in all titration programs (activated and deactivated).
When the two syringes are configured for titration, the factor is stored in the programs corresponding to the used syringe.
After the standardization, if there are more than one activated program, the user must select the titration program to be used, see page 14.



Titration

7. KF titration and data entrance

After the standarization the titration vessel is conditioned to follow with the sample analysis.

Select the titration program when there are more than one activated. The Aquamax KF Volumetric offers 2 programs for water content between 1 and 10%, one program for low content (< 1%) and one program for high water content (> 10%). The program to be used depends on the water content in the sample, see page 25.

Statistics: if statistics is desired, the number of analysis must be defined.

User: user identification (when the data output is activated).

Sample ID: Introduction of sample identification. When statistics is programmed the sample identification is common for all the analysis.

8. Vessel conditioning

The drift control is activated automatically.

When automatic drift control is programmed, on the display there is a message "PLEASE WAIT" until the drift value is equal or less than the programmed value.

When manual drift control is programmed, the user decides when the sample will be introduced into the titration vessel.

9. Sample introduction

Introduce the sample into the vessel and select "Start titration". The analysis is carried out in accordance with the selected program.

For the sample introduction use pipette if it is liquid, plastic syringe if it is like a paste and an weighing boat for solid samples.

The sample amount used in the analysis depends on the used titrant reagent and the expected water content in the sample, see page 25.

10. Result and drift control

Once the titration has finished the titrator activates automatically the drift control. Meanwhile the Aquamax KF Volumetric asks for the sample weight, the results are displayed or sent to a PC or a printer in accordance with the units selected in the titration program.

11. Vessel empty / Next sample

Usually it is possible to carry out more than one titration in the same titration medium. Nevertheless, after several analysis the medium must be changed. Press the key ESC during the drift control and select Empty Vessel (see Note). Push the pump tube to the botton of the vessel and the pump will automatically empties the vessel . Pull the pump tube in upper position (see page 15).

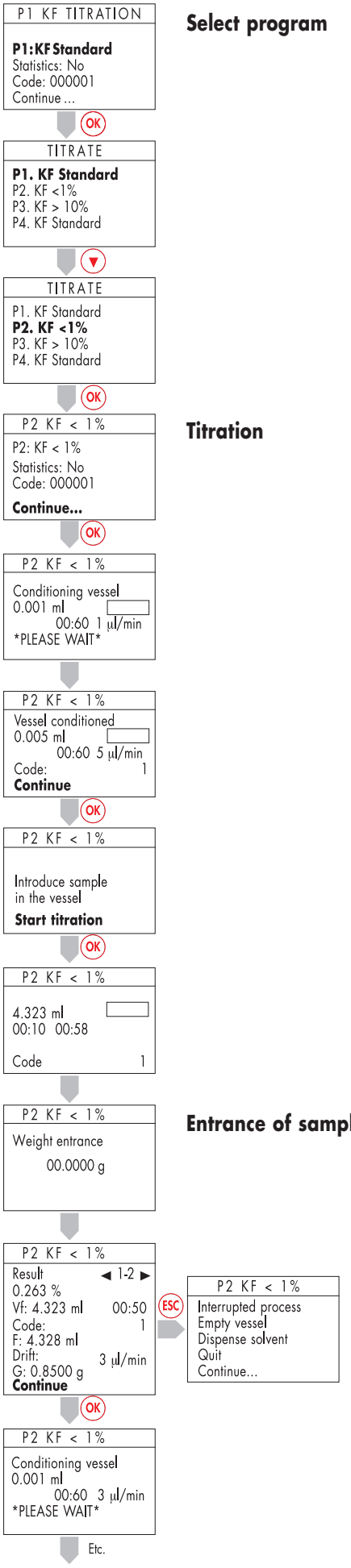
Once the medium has been changed, select *Continue* and the Aquamax KF Volumetric starts the process with the water neutralisation in the new solvent.

Note: process interrupted.

A titration can be interrupted at any moment by pressing the key ESC. On the display appears:

Process interrupted
Empty vessel
Dispense solvent
Quit
Continue

Select the desired option with the arrow keys and accept with OK.



Notes:

Stirring speed

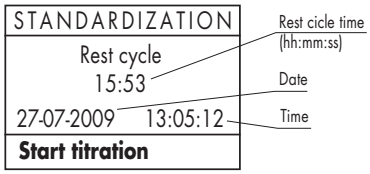
The stirring speed must be intense to ensure a rapid mixing of the added reagent and the titration medium. Insufficient stirring can easily lead to an over-titration whereas excessive stirring introduce a lot of air into the reaction medium disturbing the electrode response. The stirring speed can be modified during the vessel conditioning, pre-titration and titration.
Once the stirring speed has been selected it is recommended to maintain it constant during the analysis.

Bubbles elimination

Before starting a titration or a standardization the Aquamax KF Volumetric returns the content of the syringe inlet tube into the bottle. Afterwards the syringe loads again. This movement of the syringe eliminates the well-known problem of bubble formation in the reagent circuit.

Rest cycle

If the instrument stays in drift control more than 15 minutes it automatically activates the Rest cycle. It consists in returning the syringe reagent into the bottle, stopping the stirrer and controlling the time. The instrument waits for the user to start working again by accepting the option "Start titration". At that moment the syringe is loaded automatically, the vessel conditioning starts and the instrument calculates the drift bearing in mind the duration of the Rest cycle and the titrant used to neutralise the water present in the vessel.
If the determined drift value is correct in accordance with the programmed value, the instrument allows the sample introduction. Otherwise the drift control continues until the vessel is conditioned.



Standardization

Water standards

STANDARD (V): a solution with a known amount of water per ml of standard. The Aquamax KF Volumetric presents as water amount 5.00 mg/ml, with possibility to modify this value in accordance with the standard certificate. It is recommended to use in the standardization between 1 and 5 ml of standard.
STANDARD (G): a solution with a known amount of water per gram of standard. The Aquamax KF Volumetric presents as water amount 10.00 mg/g, with possibility to modify this value in accordance with the standard certificate. It is recommended to use in the standardization between 0.5 and 2 g of standard.
H_2O: Water. The recommended amount to be used is between 10 and 25 mg.
TARTRATE: Sodium Tartrate 2-hydrate. A primary standard with water amount of 15.66%. The amount used in the standardization should be between 0.1 and 0.15 g.
The standard amounts mentioned above correspond to titrant consumption (factor 5) between 20 and 100% of syringe volume.

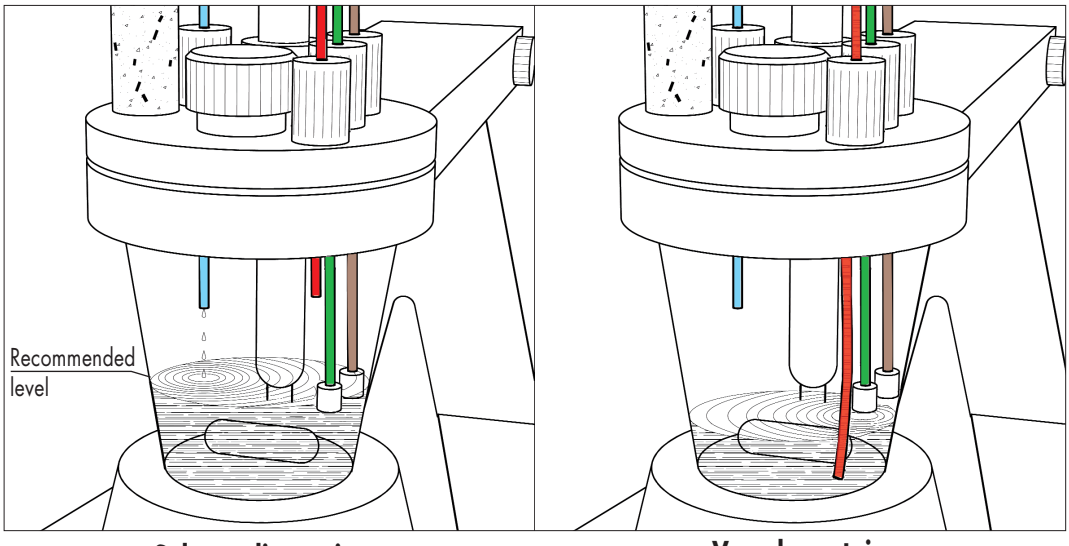
Solvent dispensing and vessel empty

Important: Introduce the aspirating tube until the vessel bottom is reached. Once the vessel is empty, place the pump on its initial position.

If the sample is solid and does not dissolve in the titration medium, hence it may possibly block the emptying pump tube. In this case, it is better to empty the vessel manually.

During the analysis (standardization or titration) the pumps tubes should not touch the titration medium.

At the end of the working day leave the vessel empty.



Solvent dispensing **Vessel emptying**

PROGRAMMING: STANDARDIZATION

By this option the user adapts the program parameters to his working method.

KF Standardization

The programing parameters are the following:

Type of titration

CLASSIC: suitable for titrations with standard reagents.
SPECIAL: suitable for titrations with special reagents for aldehydes and ketones. Recommended end point: 200 mV.

Water standard

STANDARD (V): a solution with a known amount of water per ml of standard.
The standard adition can be carried out automatically using the 2nd syringe for the standard dispensing in the titration vessel.
STANDARD (G): a solution with a known amount of water per gram of standard.
H_2O: Water.

TARTRATE: Sodium Tartrate 2-hydrate. A primary standard with water amount of 15.66%.

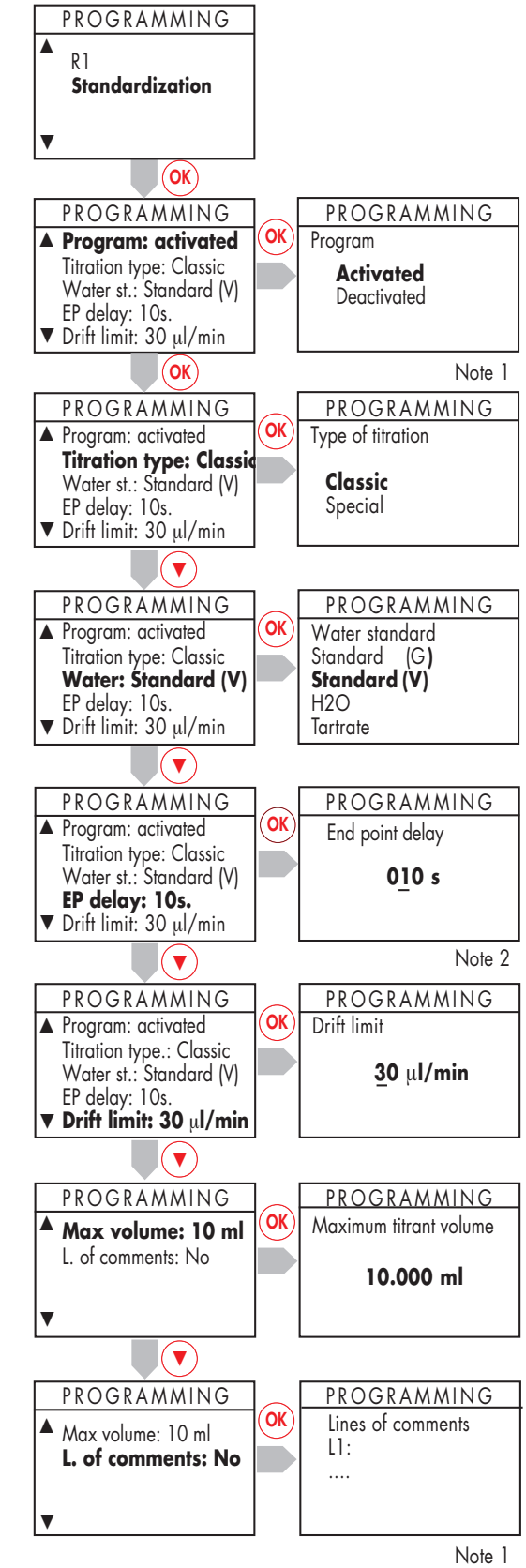
End Point Delay: *10 s.* A period of time during which the reaction end point must remain stable, an indication of the titration end.

Drift limit: 30 µl/min
In a standardization the drift control is always automatic. The user can program the drift limit. The instrument does not consider the titration medium ready for titration until the drift limit is equal or less than the programmed value.

Maximum titrant volume: *10 ml.* Security volume of the titration. If the maximum titrant volume is reached before the end point, the titration is stopped automatically and an error mesage appears "Maximum volume reached".

Lines of comments:
- *NO.*
- *YES.* Using a standard PC keyboard up to 8 lines can be introduced at the end of the program. They can be seen when the program is printed.

The determined Factor is stored automatically in all titration programs corresponding to the used reagent.



Note 1: Select with ▲ ▼ and accept with OK

Note 2: Select with ◄ ►, modify with ▲ ▼ and accept with OK

KF Titration

The Aquamax KF Volumetric offers 4 titration programs per each syringe when both of them are configured for titration.
P1 KF Standard.
P2 KF < 1% H2O.
P3 KF > 10% H2O.
P4 KF Standard.
These programs are optimized in accordance with the water content in the samples.
The programming parameters are the following:

Type of titration

CLASSIC: suitable for titrations with standard reagents.
SPECIAL: suitable for titrations with special reagents for aldehydes and cetones. Recommended end point: 200 mV.

Water extraction

BEFORE TITRATION: programming a period of time from the sample introduction in the titration vessel to the start of the titration. Suitable for samples that release the water slowly.
DURING TITRATION: programing the minimum titration duration required to guarantee the complete water extraction from the sample.
NO. The titration starts immediately after the sample introduction in the reaction vessel.

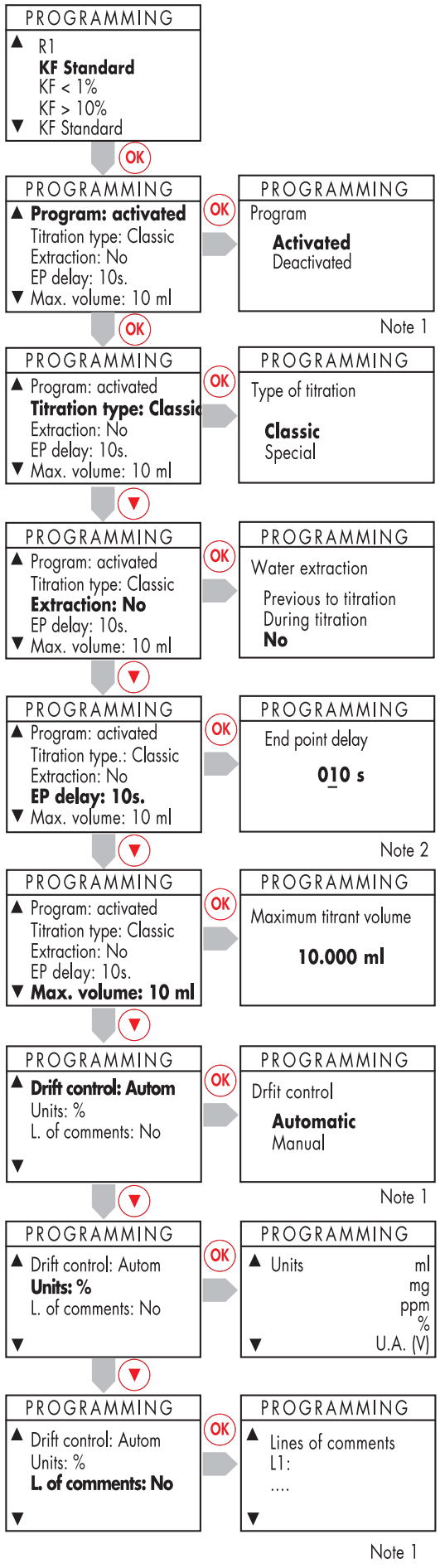
End Point Delay: *10 s*. A period of time during which the reaction end point must remain stable, an indication of the titration end.

Maximum titrant volume: *10 ml*. Security volume of the titration. If the maximum titrant volume is reached before the end point, the titration is stopped automatically and an error mesage appears "Maximum volume reached".

Drift Control:
AUTOMATIC, with limit of 30 µl/min. The limit can be modified. The instrument does not consider the titration medium ready for the sample introduction until the drift is equal or lower than the programmed value.
MANUAL. The user decides when the sample will be introduced in the reaction vessel to start the titration.
If the instrument stays in drift control more than 15 minutes (this period of time can be modified, see System), the process is automatically interrupted and the titrator switches to "Rest cycle". See page 15.

Units: Select the units to express the analysis results: ml, mg, ppm, %, A.U. (V), A.U. (G). See Units page 21.

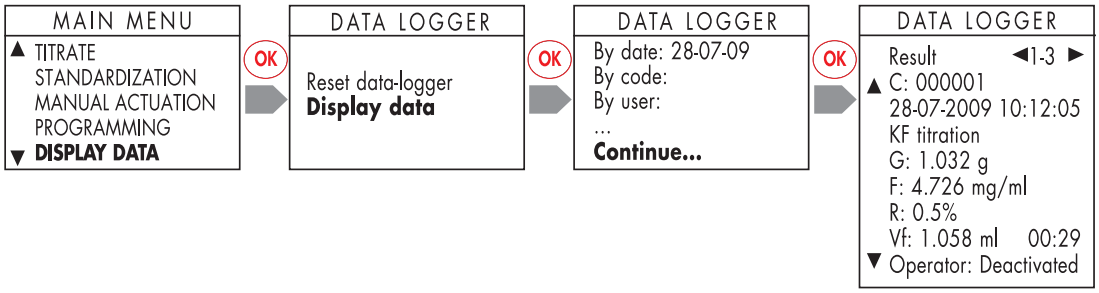
Lines of comments:
- *NO*.
- *YES*. Using a standard PC keyboard up to 8 lines can be introduced at the end of the program. They can be seen when the program is printed.



Note 1: Select with ▲ ▼ and accept with OK
Note 2: Select with ◄ ►, modify with ▲ ▼ and accept with OK

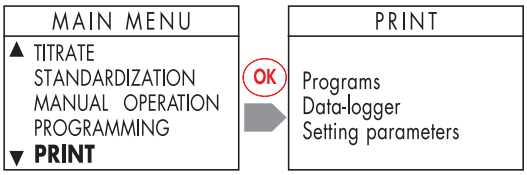
DISPLAY DATA (DATA-LOGGER)

This option allows to manage the Data-Logger.
This is a data base of up to 55 titration or standardization results. These results can be displayed, printed or sent to a PC.
The Data-logger offers different queries that allow to select the results according to a pre-set search conditions. If no condition is entered, all available data will be displayed.
When the Data-logger is full, a new result will automatically replace the oldest one.
To erase all stored results, select Reset data-logger.



PRINT

This option appears only when the Printer is activated in the Data Output. It allows to print the programs, the setting parameters and the data logger.
When a Printer or a PC are connected to the Aquamax KF Volumetric, after a titration or standardization the following data will be printed or sent to a PC:
Header, date and hour, reagent factor, drift value, sample code, titration result, final volume, titration duration, user name.



SYSTEM

This option allows to adapt the Aquamax KF Volumetric configuration to the user needs.

2nd syringe function: It allows to configure the 2nd syringe as a titrating syringe or as a dispensing syringe of water standard.

Reagent's name (10 characters): Introduction of reagent's name through an external PC keyboard. The name will appear on the printed report.

Operator's name (17 characters): Introduction of up to 4 operator's name through an external PC keyboard. The selected name will appear on the printed report.

Data output: To a printer, computer or deactivated.
Select deactivated if there is no printer or PC connected.
If there is a printer, select "Printer" and the model, thermal or dot-matrix.
Selecting "Computer" the options "Capture" and "TiCom" appear. Select the corresponding software.
If "Printer" or "Computer" is selected, the following options will appear in System:
- Report header: 2 lines of 20 characters for the company name, etc. Use an external PC keyboard.
- Sample ID: Manual introduction, via external keyboard for each sample, or automatic and consecutive assignment by the instrument (9 characters).

Test volume: Specific program for calibration of the syringe volume.

Weight entrance: Automatic or manual. If automatic weight entrance is selected, the titrator requires the balance communication protocol: baud rate, word length, parity, stop bits.

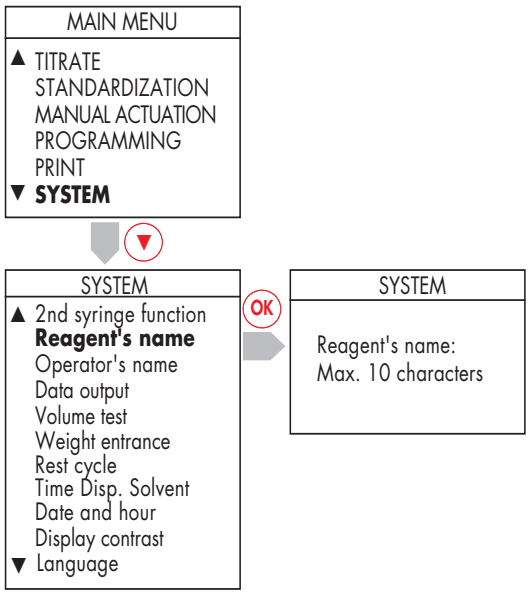
Rest cycle: It allows to modify the period of time before the start of the cycle (factory setting 15 min.). See page 15.

Solvent Dispensing Time: Time for automatic solvent dispensing at the beginning of standardization and titration, see pages 13-14.
Factory setting 13 s.

Date / Hour: Clock and calendar.

Display contrast: It increases or decreases the display contrast.

Language: English, Spanish, Italian, French.



During titration

Factor value higher than 10 mgH_2O/ml

The factor of the commercial reagents varies between 1 and 5 mg/ml. Too high values are an indication of an anomaly or an error of standard weight entrance.

High drift

This message appears when the drift is higher with 30 µl/min. than the value introduced in the program.

Drift too high

This message appears when the drift is higher with 60 µl/min. than the value introduced in the program.

Conditioning not possible

If the message "drift too high" is shown during 5 consecutive minutes the process will be interrupted. Review the titration vessel.

See Appendix 3.

Maximum volume reached

The security volume has been reached without arriving to the titration end point. The titration is interrupted automatically.

- - - - Value not calculable

The result is out of the accepted limits.

Messages in Data-logger

Attention
Data logger empty
Continue
It appears when there are not any results stored or when the search conditions do not correspond to any stored result.

Attention
All data will be erased
Continue
It appears when the option "Erase data-logger" is selected.

Other messages

Printing error
Attention
Repeat
No printout
Quit

The printer has been configured, but it is not connected or not switched on.

PC not connected
Attention
Repeat
No sending
Quit

The PC has been configured, but it is not connected or the connection does not work.

Attention
No program has been defined
Quit

It appears when all the program are deactivated.

UNITS

The result calculation is carried out in accordance with the following equations:

KF Titration. Result units

ml= ml - b
mg= (ml-b) · F
ppm= (ml-b) · F · 1000 / G
%= (ml-b) · F · 0.1 / G

Arbitrary Units (A.U.)
A.U. in Volume: (ml-b) · F · K / V
A.U. in Weight: (ml-b) · F · K / G
where:
ml: titrant volume (ml) used in the titration.
F: factor (mg/ml) obtained in the standardization.
V: sample volume (ml).
G: sample weight (g).
K: constant

Standardization

Standard	Factor calculation
In weight	F= G · X (mg/g) / ml
In volume	F= V · X (mg/ml) / ml
H2O	F= H_2O weight (mg) / ml
Tartrate	F= (Tartrate weight(mg)·15.66/100) /ml

where:
G: standard weight (g).
V: standard volume (ml).
X: water amount in the Standard. In weight expressed in mg/g and in volume in mg/ml.
ml: titrant volume (ml) used in the standardization.

STATISTICAL CALCULATIONS

Symbol	Equation
n	
x	x= ∑xi / n
SD (s)	SD= {∑(xi / n)2 / n-1}1/2
RSD (sr)	RSD= SD . 100 / x

where:
n: number of samples.
x: mean value.
SD (s): standard deviation.
RSD (sr): relative standard deviation.

To obtain the statistical data of an analysis it is necessary to repeat it as many times as number of samples are programmed, when performing a titration or standardization.

Statistical calculations in a standardization

When a Standardization with statistics is performed, the Aquamax KF Volumetric presents every result included in the statistics and allows to repeat those results which are considered as incorrect.
With the new data the statistics is calculated again and accepting the result, the Factor is stored automatically in all titration programs.

Statistical calculations in a titration

The instrument shows the number of analysis, the mean value, the standard deviation and the relative standard deviation. The Aquamax KF Volumetric does not give the option to replace the incorrect values in titration statistics.

RS 232 C INTERFACE

The Aquamax KF Volumetric has 2 serial ports RS 232 C that allow to communicate with a balance and with a printer or a PC.

Connection Aquamax KF Volumetric - Balance

To receive the sample or standard weights directly from the balance, without manual transcription.

To establish the communication:

- connect the Aquamax KF Volumetric to the balance. The cable depends on the balance pinout.
- program the same communications protocol in the balance and in the Aquamax KF Volumetric. In the Aquamax KF Volumetric, it must be selected from System, Weights entrance: Automatic.

Data output (to printer or PC)

It allows to send, through the RS 232C, the information of the results, programs, etc.

The communications protocol of the RS channel is:

Baud Rate:	9600 bps
Word length:	8 bits
Stop bits:	2 bits
Parity:	None

To establish the communication with the printer, it must be connected to the Aquamax KF Volumetric and select in System, Data output: to printer.

To establish the communication with a PC it is necessary to connect the PC to the Aquamax KF Volumetric with the corresponding cable and select System, Data output: to computer.

- Capture.
- TiCom.

An example of printed report

```
Quality Control
Ampthill
========================================
AQUAMAX KF                    SN:91901 V2.2
Wednesday, 29 July 2009              15:13
P1 KF Standard
----------------------------------------
Reagent factor:  4.703 mg/ml
Vessel drift:      5 ul/min
Sample ID: 000001
----------------------------------------
Result           Weight    End V.   Time
%                g         ml       mm:ss
------------     -------   ------   -----
       1.686     0.20720    0.743   00:24

                                Stirring  30%

Operator: Operator 1
```

```
Quality Control
Ampthill
========================================
AQUAMAX KF                    SN:91901 V2.2
Wednesday, 29 July 2009              15:10
KF Standardization
----------------------------------------
H2O content in the standard: 5.00 mg/ml
Vessel drift:      7 ul/min
----------------------------------------
Result           Standard  End V.   Time
mg/ml            ml        ml       mm:ss
------------     -------   ------   -----
       4.703       1.000    1.063   00:33

                                Stirring  30%

Operator: Operator 1
```

SPECIFICATIONS

Non-volatile memory
4 KF titration programs and 1 standardization program per syringe.
Clock/calendar
Header text: 2 lines x 40 characters
Name of up to 4 operators
Lines of comment: 8 lines x 40 characters
Up to 55 results stored in the data logger
RS communication parameters for balance

Measuring range
0.1 mg up to 100% H_2O

Results and resolution
The value of any result must be among these values:
Factor: 0 - 99.999
Result in ppm: 99999
Result in mg: 999.9
Result in %: 999
- up to 10% resolution of 0.001%
- from 10% resolution of 0.01%
Result in A.U.: 99999.9

Data logger
Storage capacity: 55 results

Display
Graphic backlit liquid crystal display,
128 x 64 dots

Keypad
Membrane, 7 keys. Keystrokes: > 6 million.
Material: PET with protector treatment.

Languages
English, Spanish, French, Italian

Stepper motor
Resolution: 1/40000 of the syringe nominal value (minimum 0.001ml)
Dispensing accuracy (as relative error) ≤ 0.2%*
Reproducibility of the dispensing ± 0.1%*
* for volumes more than 10% of the syringe volume

Syringes
5 ml TLL SL. Borosilicate glass, PE tip on the synthetic plunger.

Electrovalve
Materials in contact with the liquid: PTFE and KEL-F

Reagent tubes
Material: PTFE

Peristaltic pumps
Flow: 170 ml/min ± 15%
Tube "Novoprene", interior 3,2 mm

Inputs and outputs
Measuring electrode, BNC connector
External keyboard, miniDIN connector
RS 232C for PC or printer, telephone connector
RS 232C for balance, telephone connector
Connector RCA for stirrer

Electrical safety
Meets EC, EN 61010

EMC (Electromagnetic Compatibility)
Meets EC, EN 61326

Permitted ambient conditions
Operating temperature, 15...40°C
Storage temperature –10...50°C
<80% relative humidity, non-condensing

Power supply
External power supply 90-264 VAC, 47-63 Hz, 24 Volt DC

Enclosure
ABS and enamelled steel

Physical parameters
Weight: 4 kg approx.
Dimensions:130 x 160 x 300 mm

- At the end of the day return the reagent from the syringes to the original bottle, see MANUAL ACTUATION, page 11.

- At the end of the day empty the titration vessel, see MANUAL ACTUATION, page 11.

- If the instrument will not be in use for several days or prolonged period, clean the syringes and the pumps with methanol, see MANUAL ACTUATION, page 11. Close the reagents with their origenal screw cap. Empty the titration vessel.

- Never clean the liquid circuits, syringe and pumps with distilled water.

- Maintain the molecular sieve in the drier cartridges in good conditions.

- Verify that the aspirating tubes of syringe and pumps do not touch the bottom of the bottles. In this way the possible aspiration of sediments is avoided. They can block the tubes or damage the valve.

- Periodically verify that there are not liquid leakage on the screws of the inlet and outlet tubes of the valve and of the pumps.

- If there are crystals or other sediments inside of the syringe, take it out and clean it immediately. Any deterioration of the piston may cause leaks.

- The temperature of the liquid inside the syringe must be between 15 and 40ºC.

- The double platinum electrode does not require any special maintenance. If required, rinse with ethanol.

APPENDIX 1

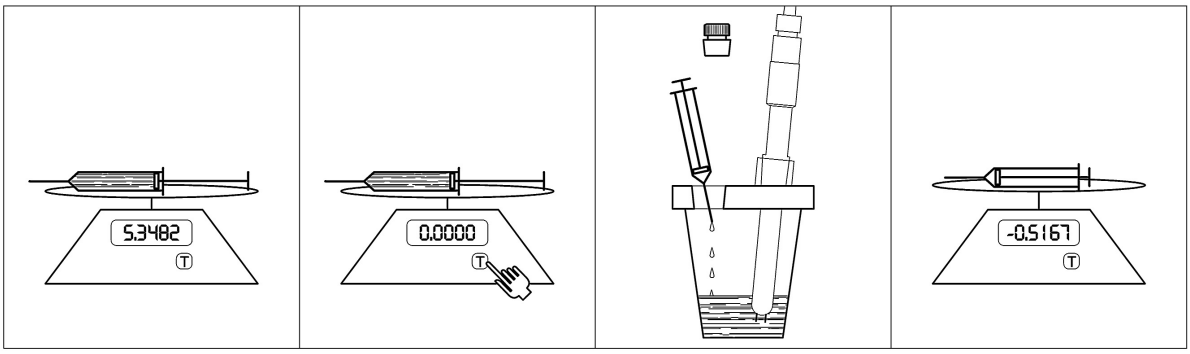
Guide for selection of titration program, sample size and reagent.

Water content (%)	Sample size (g)	Reagent Factor (mg/ml)	Titrant Consumption (ml)	Type of program
100	0.1	5	20	> 10 %
10	0.1-0.2	5	2-4	Standard
1	1	5	2	Standard
0.1 (1000 ppm)	5-10	5	1-2	< 1%

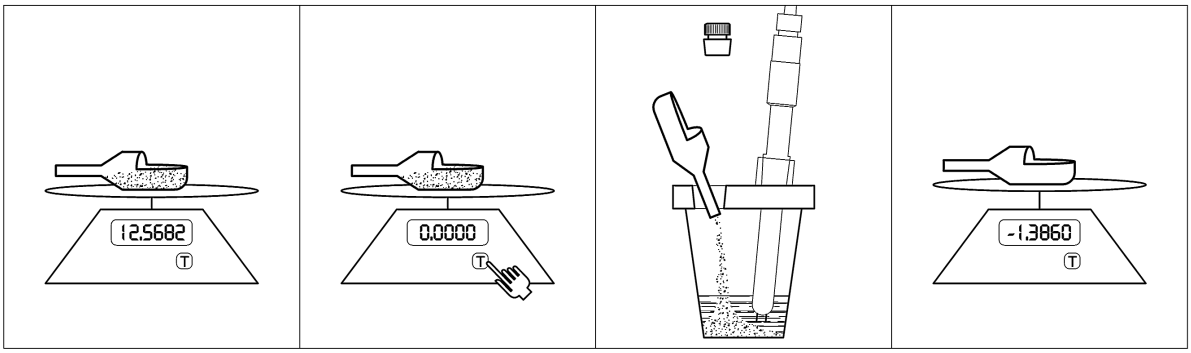
APPENDIX 2

Weight by difference and sample or standard introduction into the titration vessel.

Liquid or viscous samples



Solid samples



TROUBLESHOOTING

The message "High Drift", "Very Hight Drift" or "Conditioning impossible" appears

When setting up the instrument for the first time

- The pump and burette tubes and the syringe are impregnate of humidity.

Flush KF reagent through them to eliminate the humidity.

After the analysis of a sample

- The sample reacts with the titration medium.

Verify if the sample contains an element which reacts with the reagents. Select appropriate reagents.

- The sample releases slowly the water in the titration medium.

Increase the time of the previous extraction in the titration program.

- Secondary reaction between the sample and the titration medium.

Use reagents type K.

At any moment during the analysis

- The vessel is not perfectly closed and there is an anomaly humidity entrance.

Check the O-ring of the vessel lid. Put some grease on the elements placed on the vessel lid.

Check the correct placing of all elements on the vessel lid.

Check the molecular sieve in the drier cartridges.

The result differs from the expected value

- The sample reacts with the titration medium.

Verify if the sample contains an element which reacts with the reagents. Select appropriate reagents.

- The sample releases slowly the water in the titration medium.

Increase the time of the previous extraction in the titration program.

- The reagent factor is incorrect or it have not been determined recently.

Determine the titrant reagent factor.

- Sample with very low water content.

For samples with very low water content the reproducibility depends on the previous treatment, preparation and addition of the sample.

Increase the reagent consumption by increasing the sample amount or by using a reagent with lower factor.

- The water capacity of the solvent is exhausted.

Substite the Solvent and carry out a new titration.

Poor reproducibility of the results

- Sample size very small.

Increase the sample size, see page 25.

- No homogeneous sample

Homogenize the sample and if it is possible increase the sample size, see page 25.

- Water content very low.

For samples with very low water content the reproducibility depends on the previous treatment, preparation and addition of the sample.

Increase the reagent consumption by increasing the sample amount or by using a reagent with lower factor.

Very long titration duration. Very slow titration

- The sample does not release rapidly the water.

Increase the time of the previous extraction in the titration program.

Select appropriate reagents.

Factor not reproducible or higher than the expected value

- The used standard is water.

The use of water as a standard requires practical experience due to the small amount to be used. It is recommended to use a commercial standard.

- The used standard is sodium tartrate 2 - hydrate.

The tartrate does not dissolve immediately in the titration medium. Introduce previos extraction in the standardization program.

- The used standard is a comercial one.

Check that the added standard amount is adequate. Verify that the correct standard value was introduced in the standardization program.

The medium colour after the titration tends to be brown. Overtitration.

- The electrode is dirty.

Clean softly the platinum wires with soft tissue.

Not appropriate reagents.

NOTES

Instrument supplied by:

GR Scientific Ltd
P.O. Box 242
Ampthill, Bedfordshire
MK45 5AQ
United Kingdom

Tel.: + 44 (0) 1525 404747
Fax: +44 (0) 1525 404848
E-mail: info@grscientific.com

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